1.0

1.1

1.25   1.4   1.6

4.5
2.8   2.5
3.2   2.2
3.6
4.0   2.0

1.8

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963 A

HYDROGEN ABSORPTION BY NIOBIUM

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I. INTRODUCTION

The entry of hydrogen into metals can be very advantageous, as in the case of efficient hydrogen storage materials. On the other hand, hydrogen entry into structural metals can be devastating (the phenomenon of hydrogen embrittlement being a notorious example). One mode of entry is through the gas/metal interface. This work was undertaken to further the present understanding of the processes governing passage of hydrogen through the gas/metal interface.
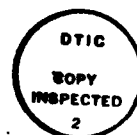
Recently, a simple model was proposed to predict hydrogen uptake by clean niobium.[1] Since it was assumed that bulk diffusion is more rapid than the surface processes,[2] the differential equations describing the change with time of the hydrogen atom fraction on the surface, θ, and in the bulk, x, could be written by summing the contributions from four reactions at the surface. Figure 1 illustrates these processes. The rate of dissociation of hydrogen gas molecules on the surface depends upon the rate of gas impingement, sticking and the availability of surface sites. This rate is expressed as $2\Gamma s_o (1-\theta)^2/N_s$, where Γ is the rate of impingement of H_2 molecules on the surface, s_o is the sticking coefficient for H_2 on a bare Nb surface, θ is the fraction of H surface sites filled and N_s is the number of surface sites per cm^2 of surface. The rate of the reverse reaction, recombination of two surface atoms to form an H_2 molecule, is expressed as $K\theta^2$, where K is a rate constant. Penetration of H atoms into the bulk proceeds at a rate proportional to H surface concentration. This rate is expressed as $\nu\theta$, where ν is a rate constant. Depletion of bulk hydrogen occurs at a rate dependent

2

Figure 1: Hydrogen absorption by niobium-surface processes

$$\frac{2\Gamma S_0 (1-\theta)^2}{N_s}$$

$$K\theta^2$$

$$\nu\theta$$

$$\beta(1-\theta)X$$
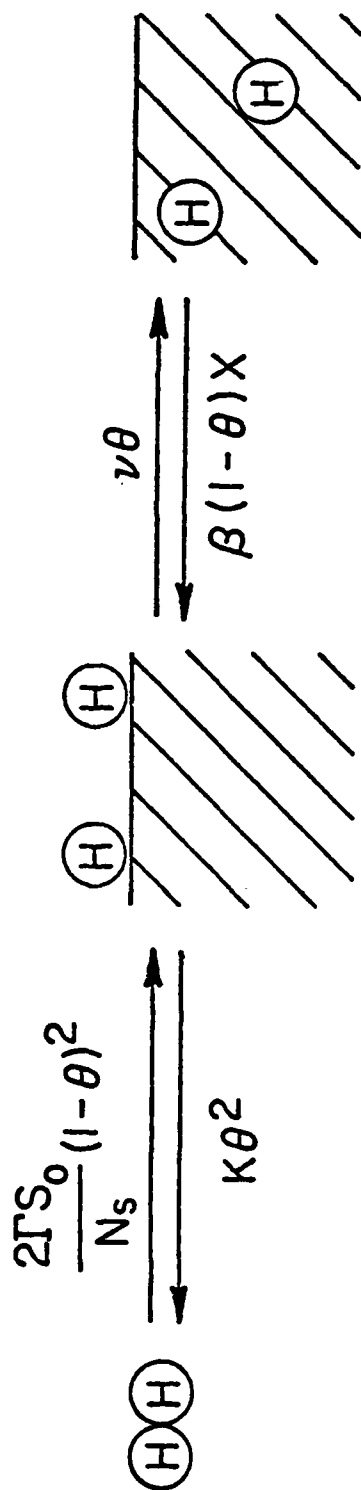
$$\frac{d\theta}{dt} = \frac{2\Gamma S_0}{N_s}(1-\theta)^2 - K\theta^2 - \nu\theta + \beta(1-\theta)X$$

$$\frac{dx}{dt} = \frac{\nu\theta}{N_l} - \beta(1-\theta)\frac{X}{N_l}$$

A

upon bulk concentration and availability of surface sites. The expression for the rate of depletion is $\beta(1-\theta)x/N_1$, where β is a rate constant, x is the atom fraction of H atoms in the bulk, H/Nb, and N_1 is the total number of Nb layers, perpendicular to a planar surface, comprising the bulk. The resulting overall rate equations are given in Fig. 1.

Two additional assumptions permitted solution of the coupled differential equations. The first is that $d\theta/dt$ is much smaller than $N_1 dx/dt$. The second is that x and θ are in quasiequilibrium, or, $d\theta/dt = dx/dt = 0$. The rate equation for hydrogen uptake which results is,

$$0.5(1-b)^2 \ln(1+Y) - 0.5(1+b)^2 \ln(1-Y) - b^2 Y = at, \qquad (1)$$

where a and b are fitting parameters and $Y = x/x_{max}$. The subscript "max" refers to the equilibrium values for a given temperature and pressure. Implicit in this model is the existance of a single type of surface site from which dissolution or desorption occurs.

Pick and coworkers[1,3] have applied this model to the H_2/Nb(110) system. Their uptake measurements, for times not exceeding 1.5 minutes, fit quite well to curves of the form of the rate equation (Eq. 1). From the dependence of the fitting parameters, a and b, on temperature, they were able to extract values for the enthalpies of hydrogen in its various states. Most interesting was the finding of Pick that hydrogen chemisorption on Nb(110) is an activated process with a barrier height, E_a, of 1.27 kcal/mole H.[3] An energy level diagram illustrating the significance of E_a in the absorption process is presented in Fig. 2. For the case shown, i.e. $E_a > 0$, the dissociation of a hydrogen

Figure 2: Energy level diagram illustrating activated chemisorption
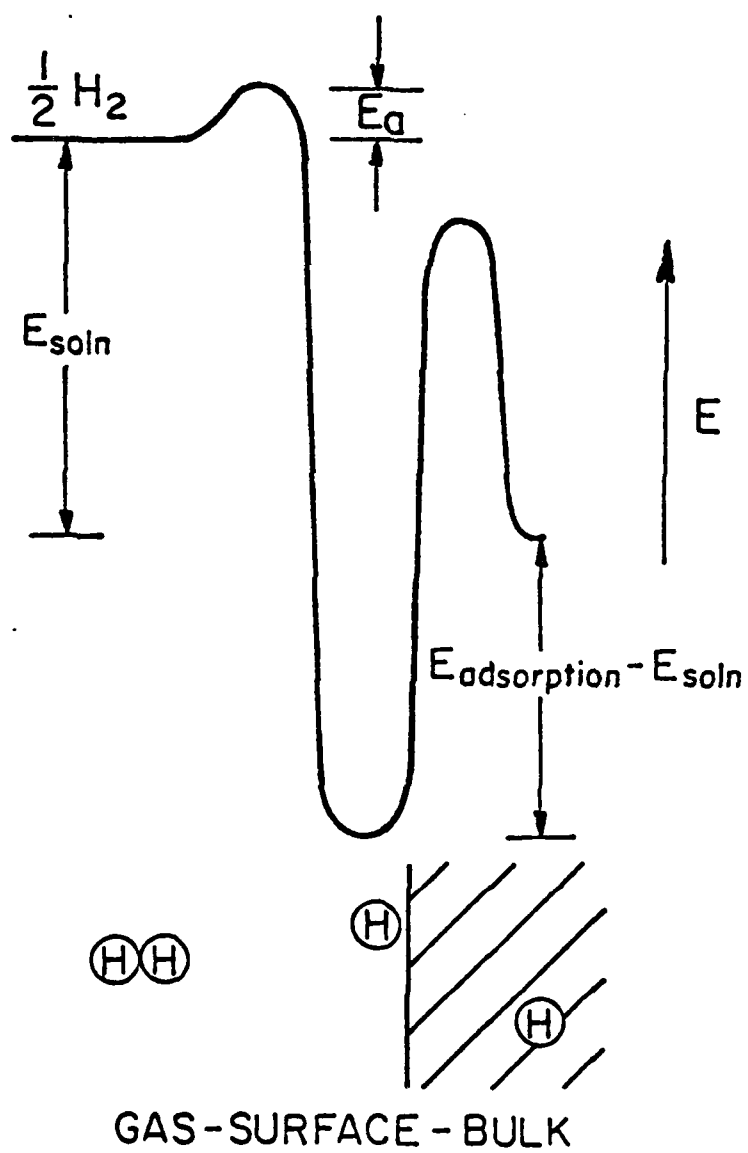
GAS - SURFACE - BULK

molecule on the surface is an activated process with an activation energy of E_a.

That the model should fit the data is not a totally expected outcome. The assumption of a quasiequilibrium between θ and x is quite reasonable. Fast surface/bulk equilibrium has been observed by Smith[4] in his photoemission studies between 390 and 600 K, Hagen and Donaldson[5] in their work function measurements over the range 140-300 K and Ko and Schmidt[6] in their flash desorption experiments between 78 and 800 K. Another assumption, not so generally accepted, however, is that there is a single surface state. Some findings support the idea that there may be more than one surface state. Examples are the results of Smith[4], Johnson and Dienes[7], Hagen and Donaldson[5] and Ko and Schmidt.[6] Whether or not a second state plays any role in absorption is not known.

One must look critically at the claim of activated chemisorption of hydrogen on niobium. Bradley and Stickney[8] studied the spatial distribution of H_2 desorbed from polycrystalline Nb to determine whether or not it was an activated process. Although they never observed the ideal $\cos\theta'$ distribution for non-activated desorption, they did approach it as they attempted to free the surface of impurities. It was their belief that any activation barrier present was, in fact, due to low levels of carbon and sulfur on the surface. Other evidence for non-activated chemisorption is the observed uptake of hydrogen by Nb at 78 K by Ko and Schmidt[6] and at 195 K by Pryde and Titcomb.[9]

The purpose of this work was to construct a system for measuring the kinetics of hydrogen absorption and then to test the model proposed by Pick and coworkers[1] on polycrystalline Nb wires. Previously, the

model had undergone testing only on Nb(110) foils. The multiplicity of orientations of a wire surface would serve to determine whether or not the results of Pick are specific to the (110) surface.

The ranges of experimental parameters must be chosen to meet several criteria. Pressures and temperatures employed should insure that only α-phase Nb/H is present both in the bulk and at the surface. In addition, pressures should result in measurable absorption in a relatively short period of time so that contamination build-up can be minimized. An additional constraint on the choice of experimental pressure is the requirement that the heat of solution of hydrogen cannot be generated at a rate faster than the rate of conduction of heat away from the sample. Otherwise, an increase in sample temperature will result. Hydrogen concentrations in the bulk should be limited to less than 1 at.% so that the solution behaves ideally.[10]

II. EXPERIMENTAL METHODS

A. SYSTEM DESCRIPTION

A schematic representation of the vacuum system is shown in Fig. 3. The basic unit is an Ultek Model TNB-X UHV system, with a double-walled stainless steel bell jar to accommodate a fluid coolant. Rough pumping was performed by cryo-sorption and titanium sublimation pumps. UHV pumping was done by a 200 1/s ion pump and an Air Products UHV-202 cryopump, from which the hydrogen adsorbant material had been removed. UHV gas pressures were measured with ionization gauges. A residual gas analyzer was used to evaluate gas purity. Hydrogen was supplied through a Pd-Ag membrane leak. A viewport on a 6-inch Conflat flange allowed a direct view of the sample for optical pyrometer measurements during high temperature sample cleaning. A mechanical shield was designed to prevent accumulation of evaporated Nb on the viewport.

The sample flange is shown in Fig. 4. Its components were assembled on a single 6-inch Conflat flange for ease of installation. There are a total of twelve electrical feedthroughs. Four of these carry current to the quartz lamps for heating of the copper blocks. In addition, there are four thermocouple feedthroughs which provide feedback to the temperature controllers. The thermocouples were designed to permit use of continuous lengths of wire (Fe and constantan) from the UHV probe ends to the external cold junctions. This was accomplished by using a Ni-based braze to seal the thermocouples into the feedthroughs. Two feedthroughs are provided for the potential leads of the resistance measuring circuit. The remaining two feedthroughs are 1/4" OFHC copper rods which serve four functic . First, they provide

Figure 3: Vacuum system schematic

ULTEK MODEL TNB-X
WITH
DOUBLE-WALLED S.S. JAR

H_2 LEAK

IG

"C"

O_2 LEAK

NIG

RGA

SAMPLE FLANGE

VIEW PORT

TC

CRYO PUMP

Ti

SORPTION PUMPS

ION PUMP

TSP

Ti

Figure 4: Sample flange
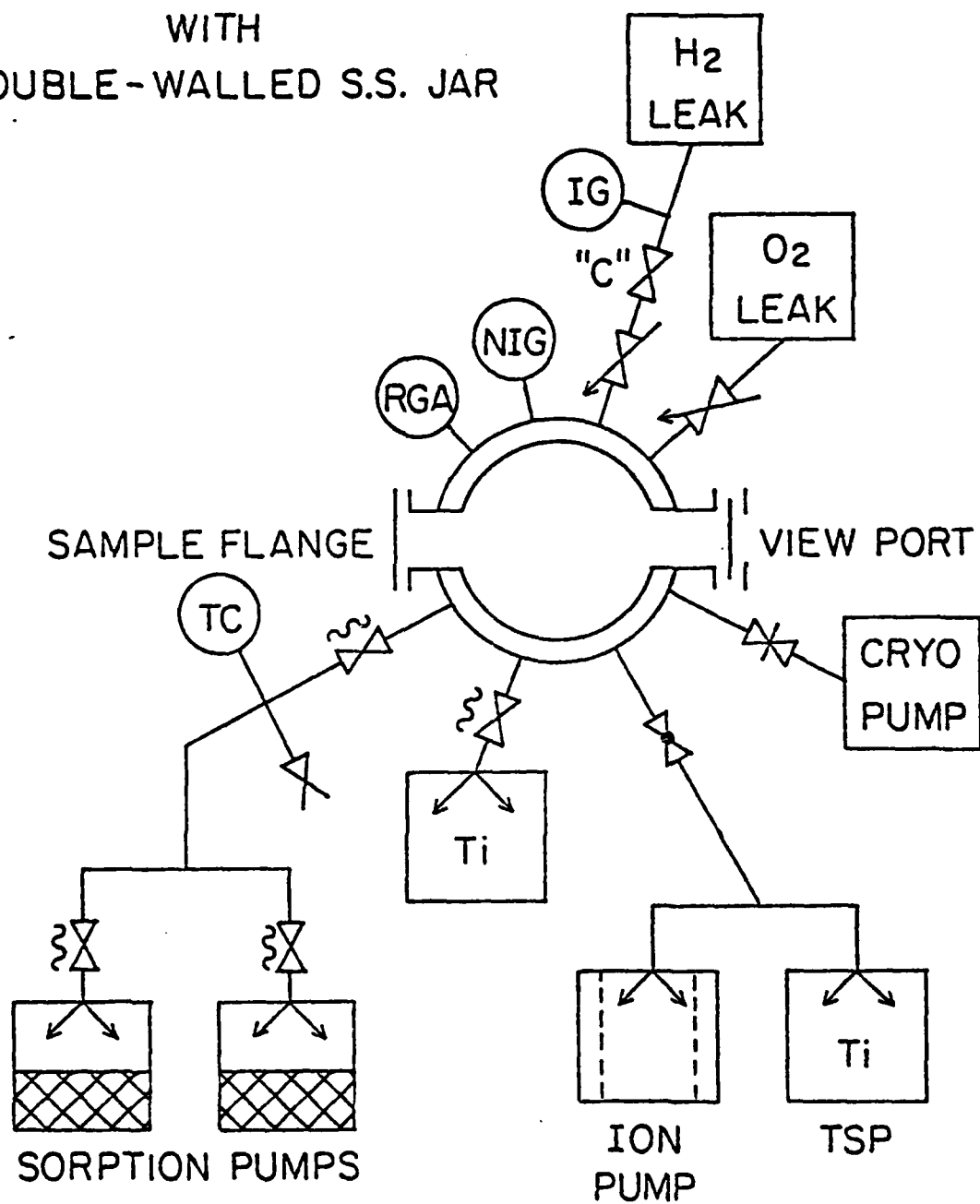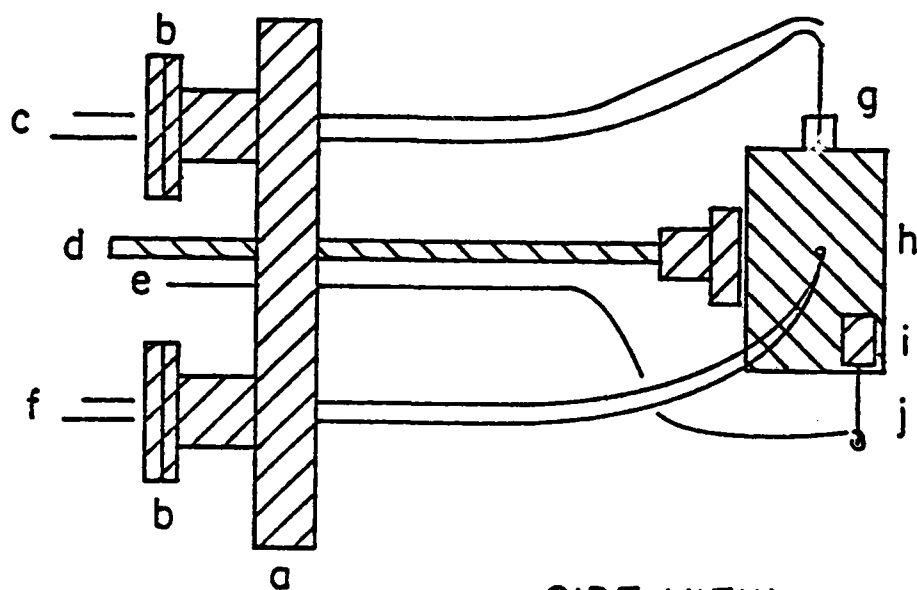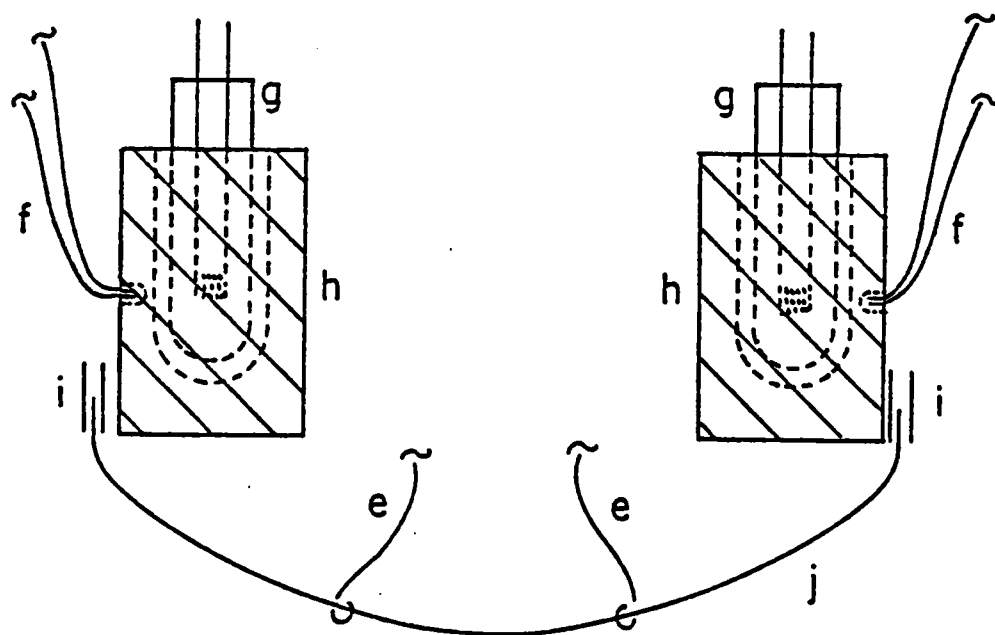(SIDE VIEW- Half of symmetrical arrangement shown)
        a 6-inch Conflat flange
        b Mini-flanges
        c Electrical feedthroughs
        d 1/4-inch OFHC copper feedthroughs
        e Electrical feedthroughs
        f Thermocouple feedthroughs
        g 150 Watt quartz lamps
        h OFHC copper hollowed blocks
        i Tungsten washers
        j Sample wire

SIDE VIEW

FRONT VIEW

supports for the sample and block assembly. They also connect to an external heat sink, composed of liquid nitrogen-cooled copper braids (this aided in temperature control). Their third function is to carry sufficient current to the sample to raise its temperature to just below its melting point for cleaning. And finally, they carry the small currents used in the 4-probe AC resistance measurements.

B. SPECIMEN PREPARATION

Samples were prepared from 0.25 mm diameter niobium wire (Teledyne Wah Chang, Albany, OR) and had lengths of about 13 cm. The results of a spark-source mass spectrographic analysis of the wire in the "as received" condition are given in Table I. Prior to use, the wires received the following treatments simultaneously: a room temperature etch for approximately one minute in a 1:1:1 lactic:nitric:hydrofluoric acid solution, several rinses in tap water, final rinses in de-ionized water and drying with nitrogen gas. Niobium potential leads of 0.05 mm diameter (Goodfellows Metals Cambridge Ltd., Cambridge, England) were similarly treated.

C. SPECIMEN ATTACHMENT TO SAMPLE HOLDER

Each end of a 0.25 mm sample was clamped to one of the thermally massive OFHC copper blocks between tungsten washers (Fig. 4). Potential leads were attached to the sample by looping the ends of two 0.05 mm wires around the sample at a separation defining the length to be monitored during absorption. This length was chosen to maximize the measured signal and to minimize the thermal gradient along the sample during the high temperature cleaning. Sintering of the leads to the

Table I: As received Nb impurity concentrations (wt. ppm)

W	< 2	Co	< 0.8	Cl	2
Ta	< 6	Fe	20	S	< 2
Sn	< 1	Mn	< 2	P	*
Zr	3	Cr	< 10	Si	20
Y	< 1	Ti	< 1	Al	7
Rb	< 0.4	Sc	< 0.3	Mg	8
Cu	2	Ca	< 15	F	9
Ni	< 1	K	5	B	< 0.2
				Be	< 0.3

* interference

Note: This analysis was semiquantitative with an accuracy of ± a
factor of three.

sample was achieved later by raising the sample temperature to 2170 C in UHV. Upon removal of the sample from the vacuum chamber, the leads could not be pulled off, i.e. sintering was successful.

D. SAMPLE CLEANING

Once the sample was sealed under vacuum, the entire vacuum system was baked at 150 C for 10-12 hours. Components were then thoroughly outgassed. The copper block assembly was outgassed at a temperature greater than that at which the subsequent measurement was to be made, while the sample was protected by its oxide coating.

1. Sample B2

The sample was decarburized at 1750 C for twenty minutes.[11] Removal of oxygen was accomplished at 2170 C for 10-20 minute intervals. These were followed by periods of cooling, until a minimum in the system pressure was observed while the sample was hot. This pressure was about 3×10^{-9} torr. Following the final cool-down period, the sample temperature was raised to 2170 C for one or two minutes. Upon cooling, the sample temperature stabilized to that pre-set on the automatic temperature controllers.

2. Sample B3

This sample was cleaned in a less vigorous manner than B2. The reason was, that by the time B2 was clean according to the minimum pressure criterion, significant grain boundary sliding had occurred, producing "hot spots" during high temperature cleaning. It was hoped that an abbreviated cleaning procedure might be adequate, and, that it

would preclude grain boundary sliding.

Decarburization took place at 1700 C for 20 minutes. Intervals at high temperature (2050-2180 C) totalling 34 minutes followed. The vacuum was about 10^{-8} torr at the end of each interval. The sample temperature was raised to 2200 C for 1-3 minutes just before each run.

E. ABSORPTION MEASUREMENTS

Measurement of the sample's resistance with exposure to a hydrogen environment was accomplished by a 4-probe AC bridge technique. The bridge circuit was originally designed for low power use with resistance thermometers at cryogenic temperatures.[12] Its low measuring current requirement (1 mA) made it well-suited for the absorption measurements since it was desirable that the measuring current's contribution to sample heating be negligible.

The off-balance bridge signal, as amplified and read at the lock-in output (Fig. 5) was calibrated and found to be linear over the range of resistance change observed during hydrogen absorption. Absorption was terminated before dissolution of one atomic percent hydrogen. In the off-balance mode, the bridge sensitivity was greater than 1×10^{-6} ohm for a specimen resistance of about 0.3 ohm.

The bridge was balanced when the sample temperature was stabilized to within 0.2 C of the set point (5-10 minutes). A chart recorder made an analog record of the lock-in output. A given hydrogen pressure in the vacuum chamber was achieved rapidly by opening the "C" valve in the hydrogen line (Fig. 3). Pressure was maintained constant by monitoring the signal produced by a nude ion gauge (with thoriated iridium filament

Figure 5: Resistance measuring circuit schematic

SAMPLE
0.3Ω

A.C. BRIDGE[12]

PRE-AMP

LOCK-IN   f = 200 Hz

CHART
RECORDER
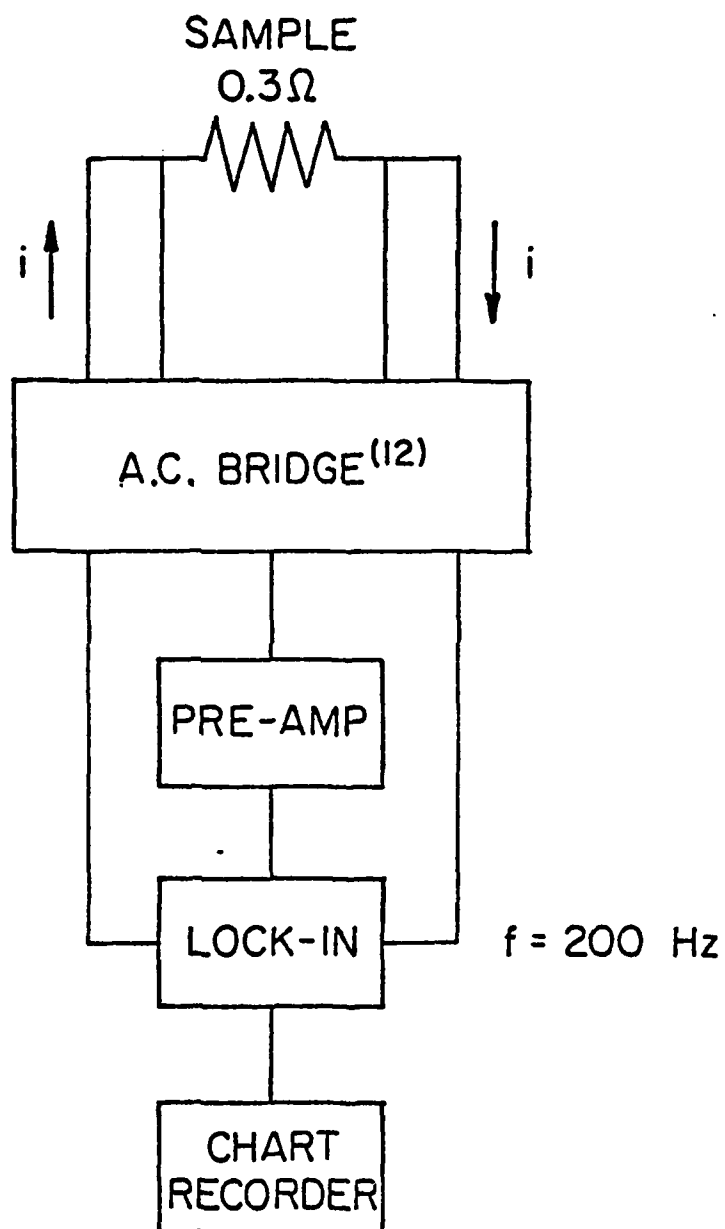
to minimize hydrogen dissociation) in the vacuum chamber, and making necessary adjustments of the conductance of the variable leak valve.

F. CALCULATION OF $Y = x/x_{max}$

Resistance measurements were converted to Y values in the following way. The solubility expression of Pryde and Titcomb[10] was used to calculate x_{max},

$$x_{max} = p^{1/2}\exp((4345\pm161)/T)/(39540\pm3380), \tag{2}$$

where p is measured in torr and T in K. The bulk fraction, x, was calculated using,

$$x = (10,000\Delta R)/(G\rho_H), \tag{3}$$

where ΔR is the measured change in resistance in ohms, G is a geometrical factor for the wire samples with units of cm^{-1}, and ρ_H is the increment in resistivity due to an addition of 1 at.% H to Nb. The value used for ρ_H was that found by Pfeiffer and Wipf,[13] 0.64 ± 0.06 $\mu\Omega$cm. The geometrical factor, G, was evaluated by measuring the sample resistance as a function of temperature over a range of 20 C, and using the relationship,

$$G = (\Delta R/\Delta T)/(\Delta\rho/\Delta T), \tag{4}$$

where $\Delta\rho/\Delta T$ is the change in resistivity of pure Nb with temperature for the same 20 C range. The extensive measurements of Nb resistivity of Abraham and Deviot[14] were used to evaluate $\Delta\rho/\Delta T$.

III. RESULTS

The R(t) curve measured for sample B2 is presented in Fig. 6 with the corresponding Y(t) values. The fit to Pick's absorption model[1] of the first 235 seconds of exposure is also plotted in Fig. 6. The fitting parameters for this fit (T = 350 K, P_{H_2} = 3 x 10^{-6} torr) are: a = 0.0017, b = 180±0.4. The "a" value corresponds to the curve's initial slope, while the "b" value determines the extent of subsequent curvature.

In Fig. 7, results for sample B3 are presented. The conditions were the same as those for the previous run except that the hydrogen pressure was a factor of one hundred greater. The Pick model fit for the first 120 seconds of exposure is also shown. To determine the reproducibility of the measurements, a total of three runs at 350 K were performed at a pressure of 2.6 x 10^{-4} torr. Curve fitting produced three pairs of identical fitting parameters (Table II). As will be discussed later, the values for higher pressure are not only related to hydrogen uptake, but to changes in sample temperature as well.

Levels of gaseous impurities in the hydrogen were measured with the residual gas analyzer. They were as follows:

$$P_i/P_{H_2} \times 100 \ (\%)$$

	He	H_2O	CO	mass 20	Other
Sample B2	.17	.07	.07	--	<.01
Sample B3	.003	.001	.003	.02	<.001

Hydrogen pressure was regulated to within 30% for pressures in the 10^{-6} torr range and to within 10% for the 10^{-4} range.

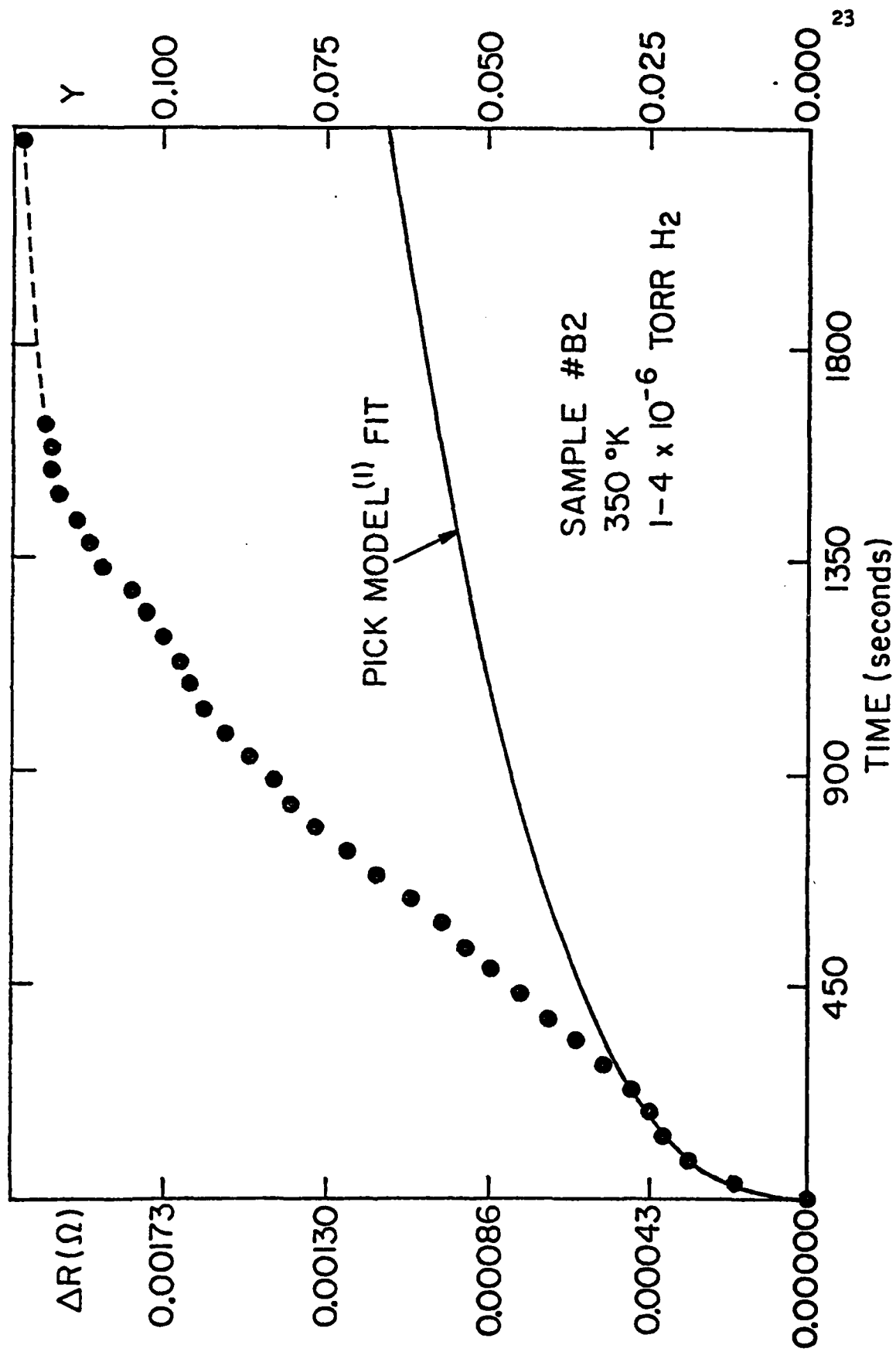Figure 6: Variation of Nb wire resistance with hydrogen exposure

23

Figure 7: Variation of Nb wire resistance with hydrogen exposure
and temperature rise due to the heat of solution
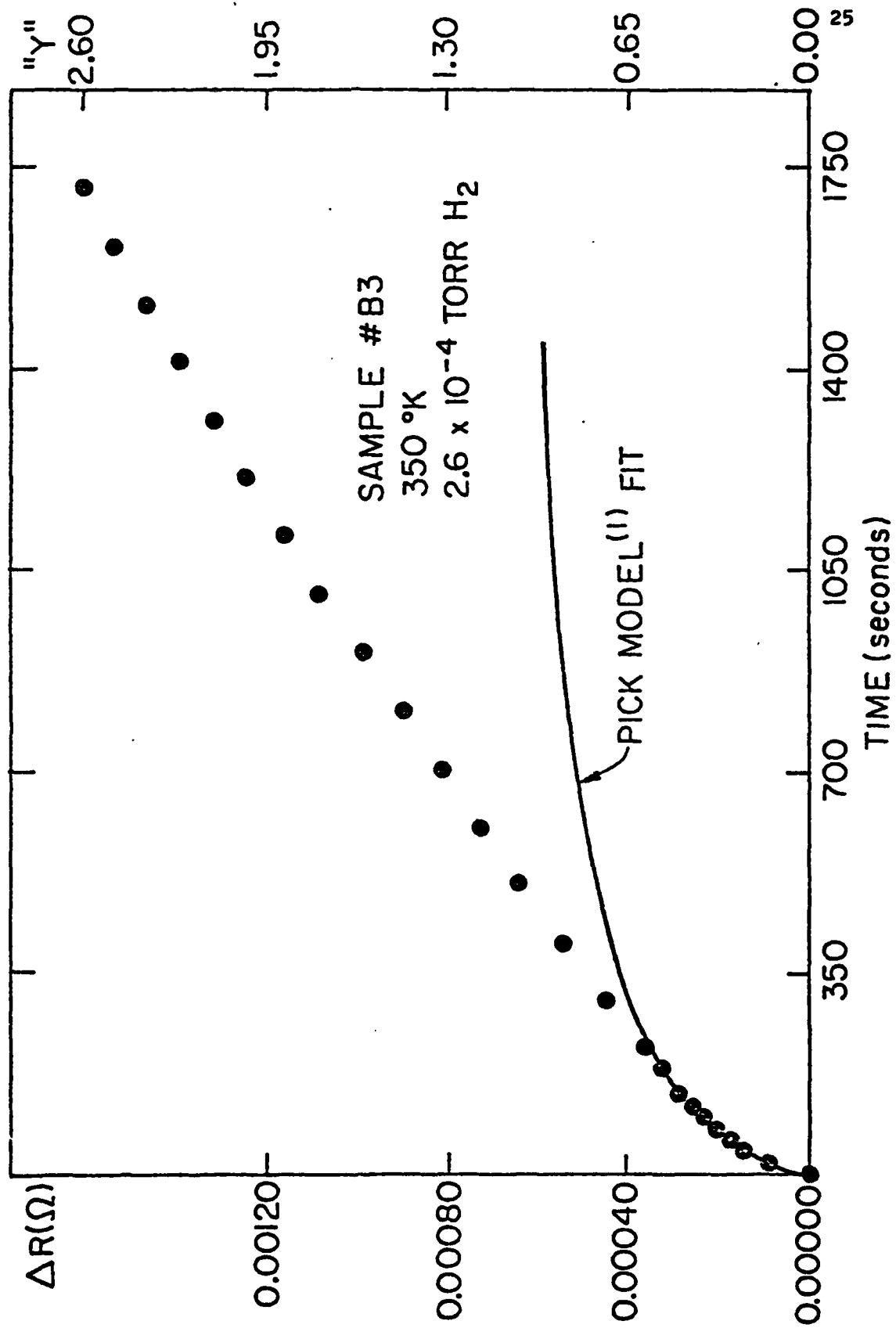
Table II: Fitting parameters

Sample B2

Extensive cleaning-grain boundary sliding

Fit for first 235 sec. of uptake

T(K)	p(torr)	a	b
350	3×10^{-6}	.0017	180 ± 4

Sample B3

Abbreviated cleaning-no grain boundary sliding

Length = 1/2 that of B2

Fits for first 120 sec. of uptake

T(K)	p(torr)	a	b
350	2.6×10^{-4}	.0188	2.50 ± 0.04
"	"	.0188	2.50 ± 0.04
"	"	.0188	2.50 ± 0.04

The absolute temperatures measured by the thermocouples were estimated to have an accuracy of ±2 K and the temperature regulation was ±0.18 K.

Upon removal from the vacuum system, a wire with a history similar to that of sample B2 was examined at a magnification of 450X in a JSM-25S scanning electron microscope. No evidence of gross facetting of the wire surface was found.

IV. DISCUSSION

As indicated in Fig. 6, the form of the theoretical curve for (110) foils does seem to describe the hydrogen uptake by wire samples at short times. However, significant deviation arises at an exposure of the order of 675 Langmuirs. A linear region is observed, with uptake in excess of that predicted by the model. At longer times, the slope of the curve decreases toward zero.

The constant uptake regime agrees qualitatively with the results of Fromm and Uchida[15] for hydrogen uptake by tantalum wires. It is important to note that the deviations from the model behavior cannot be simply explained by an argument based on build-up of background contamination. If this were the case, absorption would be expected to drop, with time, below that for a clean surface. From the levels of residual contaminants in the hydrogen, one can get a rough idea of the rate of accumulation of surface contaminants. Surface build-up of X atoms from a diatomic gas, XY, in monolayers/sec, M_X, may be expressed by,

$$M_X = (p_{XY}s_X)/(\sqrt{2\pi m_{XY}kT_{XY}}\rho_{surf}) \text{ monolayers/sec}, \qquad (5)$$

where p_{XY} is the partial pressure of the gas, s_X is the sticking probability of an X atom from an impinging XY molecule (assumed to be 1.0 for a worst case estimate), m_{XY} is the molecule's mass, k is Boltzmann's constant, T_{XY} is the gas temperature (typically the system wall temperature) and ρ_{surf} is the density of surface sites which can accomodate X atoms (for the present purpose, it is assumed that the density of sites is equal to the surface density of Nb atoms on a smooth cylindrical wire using (110) atom spacing).

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For the run shown in Fig. 6, the hydrogen pressure was approximately 3×10^{-6} torr. The level of CO in the background was found to be 2.1×10^{-9} torr. Using Eq. 5, one calculates the rate of carbon contamination of the surface to be 2.0×10^{-3} monolayers/second; it will take 500 seconds before a monolayer of carbon will form on the surface. If one includes a surface roughness factor estimate, this time should increase proportionally. The oxygen accumulation rate from both H_2O and CO is calculated to be twice that of carbon. Clearly it would be impossible to say that the Nb wires studied here had clean surfaces. It was hoped that reproducibility of the data would be a test of cleanliness. However, in this study, it is possible that the contamination rate was reproducible. Future studies should incorporate an independent means for ascertaining surface cleanliness (e.g. AES).

The form of the absorption curve in Fig. 7 appears to agree with that of Fig. 6. However, it represents the observed ΔR when hydrogen uptake is rapid enough that the heat of solution raises the sample temperature. One can estimate the upper limit on useful pressures by evaluating the upper limit for conduction of heat away from the sample to the heat sinks (copper blocks). This estimate is based on the dimensions and temperatures of the sample configuration. The rate of heat input, Q_{IN}, to the wire due to solution of hydrogen can be calculated since,

$$Q_{IN} = s\Gamma\Delta H_s A, \qquad (6)$$

where s is an estimated sticking coefficient for hydrogen on Nb, Γ is the rate of impingement of H_2 molecules on the surface, ΔH_s is the heat of solution of hydrogen in Nb and A is the surface area of the wire through which hydrogen is being absorbed. Using the sample diameter of

0.025 cm, absorbing length of 4 cm, approximate sticking coefficient of 0.01^6 and heat of solution of 16 kcal/mole H_2[10], it is found that,

$$Q_{IN} = 1.2 \times 10^{-4} \text{ p kcal/sec} \tag{7}$$

with p in units of torr. Conduction of heat from the wire proceeds at a rate of,

$$Q_{OUT} = 2k'\sigma \Delta T/1. \tag{8}$$

In this expression, Q_{OUT} is the rate of heat conduction, k' is the thermal conductivity of Nb, σ is the wire's cross-sectional area, ΔT is the temperature difference between the wire center and a copper block and 1 is the distance between the wire center and a copper block. The values of these parameters are 0.54 Watt cm^{-1} K^{-1} for k'[16], 6 cm for 1 and a maximum allowable T of 0.2 K. The resulting expression is,

$$Q_{OUT} = 4.3 \times 10^{-9} \text{ kcal/sec.} \tag{9}$$

From the requirement that no net accumulation of heat in the wire can occur, or,

$$Q_{IN} = Q_{OUT}, \tag{10}$$

one arrives at the result that the maximum hydrogen pressure which will allow the sample temperature to remain constant to within 0.2 K is approximately 4×10^{-5} torr. Thus, in Fig. 7, where $p_{H_2} = 2.6 \times 10^{-4}$ torr, the Y values calculated from the ΔR's actually exceed one, since the contributions to ΔR from the heating effect of the sample were not subtracted. A direct measure of wire temperature with time would be required to correct for heating. Shorter and larger diameter wires would permit use of higher pressures without heating.

In order to calculate the effective intrinsic energy levels for wire samples, measurements of absorption must be made at a constant value of hydrogen pressure over a range of temperatures. This will make

a very interesting study and is well within the capabilities of the system designed, if the surface cleanliness can be assured.

Wire specimens have a marked disadvantage because of the high temperature grain boundary sliding observed. Polycrystalline Nb foil specimens would not be expected to show such severe sliding. Unfortunately, upon heating, they recrystallize into a single surface orientation (i.e. (110))[1,3].

V. CONCLUSIONS

The vacuum system and AC resistance measuring system described have been used successfully in the measurement of hydrogen absorption kinetics. The basic limitation of this system, when used with wire specimens, lies in the inability to carefully control and characterize the surfaces under investigation. In principle, the macroscopic study of absorption kinetics can be used to determine orientational, structural and impurity level effects. An independent means of assessing surface cleanliness would be an important supporting technique for understanding the results of these measurements.

The simple kinetic model of Pick and coworkers[1] does in fact follow the form of the curves for early uptake of hydrogen by Nb wire, but is inadequate to describe the longer time behavior observed. A complete model would have to include the attainment of steady state uptake following the initial transient period.

The heat of solution of hydrogen in Nb can be neglected in these measurements under one condition. That is, that the hydrogen pressure is chosen to limit the rate of evolution of heat to the maximum rate of conduction of heat away from the sample. The limiting pressure depends upon the sample dimensions and system configuration.

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University of Illinois, Dept. Of Metallurgy and Mining Engineering	unclassified
	2b. GROUP AD·A114813

3. REPORT TITLE

Hydrogen Absorption By Niobium

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13. ABSTRACT

The absorption of hydrogen into niobium was studied using UHV methods to control surface impurities and resistivity techniques to determine the amount of hydrogen in solid solution.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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